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AEROSPACE REPORT NO.
TOR-0091(6064)-2

Structural and Tribological Studies of MoS₂ Solid Lubricant Films Having Tailored Metal-Multilayer Nanostructures

Prepared by

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15 December 1991

Prepared for

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Wright-Patterson Air Force Base, OH 45433

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Contract No. F04701-88-C-0089

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STRUCTURAL AND TRIBOLOGICAL STUDIES OF MoS_2 SOLID LUBRICANT
FILMS HAVING TAILORED METAL-MULTILAYER NANOSTRUCTURES

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15 December 1991

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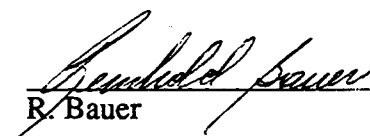
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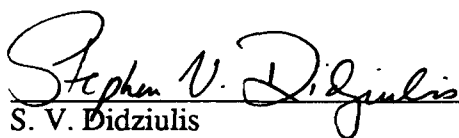
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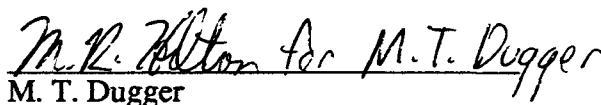
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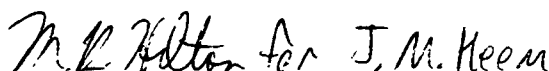
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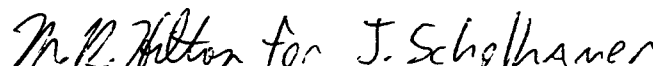
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ABSTRACT

Molybdenum disulfide (MoS_2) solid lubricant films were prepared by rf magnetron sputtering on 440C steel, 52100 steel, and Si substrates. This study concentrated on films that were multilayer coatings of MoS_2 with either Ni or Au-Pd(20%) metal interlayers. Multilayer thickness ranged from 0.2 nm to 1.0 nm while the multilayer periodic spacing ranged from 3 to 10 nm. Scanning electron microscopy and X-ray diffraction revealed that the multilayer films had dense microstructures that, in some cases, exhibited significant orientation of their basal planes parallel to the substrate. Film endurance was assessed in sliding contact by using thrust washer tests and in rolling contact by using thrust bearing tests. Some film microstructures exhibited excellent endurance. Brinell indentation indicated that the metal layers can improve film fracture toughness. Friction in air and ultra-high vacuum (UHV) was investigated using a UHV-compatible test apparatus. Friction coefficients between 0.05 and 0.08 were measured in UHV.

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ACKNOWLEDGEMENTS

The authors would like to thank P. Adams for performing the XRD experiments, J. Childs for assistance with some of the SEM and indentation measurements, J. R. Lince for interpretation of the EXAFS data at Aerospace, E. H. Sorroche (Sandia) for performing the pin-on-disk friction measurements and taking the in-situ Auger data, and D. R. Tallant (Sandia) for performing the Raman measurements.

This work was supported at Aerospace by the Air Force Systems Command, Space Systems Division, and the Strategic Defense Initiative Organization under Contract F04701-88-C-0089; and at Sandia National Laboratories by the U. S. Dept of Energy under Contract DE-AC04-76-DP00789. The authors also wish to thank B. D. McConnell, K. R. Mecklenburg, L. J. Fehrenbacher, P. D. Fleischauer, and L. E. Pope for helpful discussions, as well as D. Polk of the Office of Naval Research for providing initial support to Ovonic for MoS₂ multilayer depositions under Contract N00014-87-0273, and to Prof. B. Kramer (George Washington Univ.) for important initial discussions with Ovonic on MoS₂ film growth issues.

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I. INTRODUCTION

Sputter-deposited films of molybdenum disulfide (MoS_2) have been used as lubricants for over twenty years in space and vacuum applications because of the material's low friction, negligible vapor pressure, and temperature insensitivity (relative to liquid lubricants).¹ MoS_2 films have been used extensively in sliding applications, such as release mechanisms, pivoting hinges, and telescoping devices. There is an increasing interest in using MoS_2 films in rolling applications, such as precision bearings.²

Structural studies of sputter-deposited MoS_2 have often reported a porous, columnar-plate morphology, sometimes with a dense underlayer near the substrate.^{3,4,5} When reviewed in aggregate, X-ray diffraction (XRD) and scanning and transmission electron microscopy (SEM/TEM) investigations indicate that the columnar morphology is a direct consequence of the anisotropic crystal structure of MoS_2 , which causes significantly different reactivities on different facets of the compound.^{6,7,8} MoS_2 is composed of hexagonal-packed planes consisting of a layer of Mo bounded on each side by a layer of S. All effective strong bonding is within the resulting "sandwich", not between adjacent S layers. The (001) basal planes have low shear strength and provide an atomic mechanism for plastic deformation, which allows MoS_2 to function as a lubricant. The (001) basal surface of MoS_2 is unreactive relative to edge plane surfaces such as the (100) and (110) facets. The latter provide the reactive sites on MoS_2 catalysts.⁹

These various facets have different growth rates during deposition. The edge plane surfaces grow faster than the basal surface, as evidenced in TEM lattice imaging, by the formation of anisotropic plate-like islands in which the (002) basal planes are parallel to the flat surfaces of the plates.⁸ Near the interface, the islands have two general orientations: (1) an "edge island" in which the (100) or (110) edge-plane surface is parallel to the substrate, or (2) a "basal island" in which the (002) basal plane is parallel to the substrate. Competitive growth occurs such that the edge islands evolve into columnar plates, which, in turn, shadow and inhibit the continued growth of basal islands. This competitive process results in the growth of a porous, columnar-plate microstructure having (100) and (110) preferred orientation parallel to the substrate.

The porosity that develops over the shadowed basal islands can affect tribological performance. Such voids collapse during contact and convert a large fraction of the total film thickness into film debris early in wear (which is detrimental in debris-ejecting applications, such as ball bearings).^{3,5,10,11} Denser morphologies can be formed by retarding or eliminating edge island growth, which has been accomplished by adjusting deposition parameters (lowering pressures,^{4,12,13} bombarding with ions during growth^{14,15}) or by including dopants (water vapor in the ambient,^{11,16} or 7 to 10% nickel in the target¹⁷).

Chemical modification or passivation of the substrate surface can suppress initial edge island formation by eliminating surface chemical sites that bond to the reactive edge facets.⁷ However, growth defects, such as crystal curvature, cause edge islands to evolve within 10 to 30 nm of the interface.^{8,18} A passive surface could be controllably reimposed during film growth to form a multilayer film with periodic spacing of the imposed layers. Edge island formation, columnar growth, and porosity might then be avoided. In this paper, we report the first investigation of the properties of Ni- MoS_2 and Au-Pd(20%)- MoS_2 multilayer films.

II. EXPERIMENTAL PROCEDURES

The majority of the films were prepared by rf magnetron (RFM) sputtering using a cylindrical chamber (base pressure: 1×10^{-5} Pa) having a vertical four-target configuration. In these runs, one metal [Ni or Au-Pd(20%)] and one or, in later runs, two MoS₂ target were used. The substrates were mounted vertically to the surface of a carousel that rotates on an axis coincident with the axis of rotation of the chamber and that moves the substrates from in front of one target to another. Use of several carousels with different diameters allows the substrate-to-target distance to be varied. The size of the carousels isolates the substrates from the different deposition fields. The individual layer thicknesses were controlled by varying a combination of the deposition rate (target power), carousel bias level, and carousel rotation rate. The total film thickness was controlled by the total number of carousel rotations. The deposition pressure was 0.266 Pa and sample temperature never exceeded 100°C.

One set of films was prepared by ion-beam-assisted deposition (IBAD), using a chamber (base pressure: 3×10^{-6} Pa) in which sputter targets are rotated in front of a 3-cm-diameter neutralized argon beam produced by a standard Kaufman-type source. The angle between the ion beam axis and the target normal was 45°. The substrates were mounted parallel to the target on a rotating, cooled holder. Layer thickness and spacing was controlled by beam current, voltage, and duration on each target. The beam was shut off while alternating targets were rotated into position.

Substrates in each deposition run included 440C steel blocks (Rockwell C hardness 58-60, polished to 100 nm finish) used for sliding wear tests and XRD measurements, 440C steel friction measurement disks (Rockwell C hardness 58-61; final polish to 0.3 μ m grit), 440C or 52100 steel thrust bearing raceways (INA W 1-1/2 thrust bearings), Si (100) wafers, and in some cases Kapton films. Three groups of depositions were performed. Group I used 52100 bearing raceways, while Groups II and III used 440C bearing raceways and included Kapton films. However, the multilayer films in groups II and III had a 50-nm overcoat of pure MoS₂ to facilitate initial film transfer to the uncoated counterface (see Section III, RESULTS). Total film thickness was nominally 1 μ m in both groups. The following multilayer thicknesses (as confirmed by XRD) were prepared: Group I: pure MoS₂, 0.3nm-Ni/4.8nm (the first number is the metal thickness, and the latter number is the periodic spacing of the metal multilayers), 0.5nm-Ni/5.1nm, 0.8nm-Ni/5.0nm, 1.0nm-Ni/5.1nm, 0.2nm-Ni/3.3nm, and 0.8nm-Ni/3.9nm; Group II: 0.7nm-Ni/10nm, 0.9nm-Au/10nm; Group III: pure MoS₂, 0.7nm-Ni/10nm, and 0.9nm-Au/10nm. The pure MoS₂ films in Group III had 2-nm Ni interlayers deposited at the film-440C steel interface to promote adhesion. The pure MoS₂ films in Group I had no metal interlayer deposited at the film-52100 steel interface. All of the multilayer films had metal (Au or Ni) interlayers at the steel-film interface. The IBAD films had thicknesses of 1.5nm-Au/3.6nm, and were deposited only on 440C steel wear blocks. For comparison purposes, films previously produced by rf sputtering at 70°C ambient temperature (RF-AT) and at 2.66 Pa sputter gas pressure were used as characterization references.^{5-8,10,11} Figure 1 illustrates the typical multilayer structure.

Structural characterization was performed by XRD. The periodic spacing of the multilayer structure was determined by grazing angle XRD using Cu K α radiation following calibration runs and after substrate coating runs. The precision of this method, often within a few hundredths of a nanometer, is strongly related to the number of orders of reflection observed. Actual multilayer

spacings are inferred from comparison of the locations of all order peaks with predictions based on model calculations.¹⁹ Preferred orientation of the films was determined by XRD using a vertical powder diffractometer equipped for normal θ - 2θ scans using Cu K α X-rays. This instrument also detected multilayer peaks. The X-ray scattering vector G (the vector subtraction of the incoming X-ray vector from the outgoing X-ray vector) was always parallel to the surface normal.⁶

Film morphology was examined by SEM using procedures reported elsewhere.⁵ Cross-sectional samples were prepared by Brale indentation, which also provided an indication of interfacial fracture toughness.¹¹ Sliding wear endurance tests were performed using a thrust washer on a coated flat in an apparatus described elsewhere.¹⁰ Thrust bearing endurance tests were conducted at 2500 rpm under a mean Hertzian stress of 0.48 GPa (70 ksi) in a dry nitrogen environment. The test was terminated when measured reaction torque exceeded ten times the running torque. Friction tests were run in an instrument²⁰ in room air (25 to 35% humidity) or under UHV conditions (6.7×10^{-7} Pa). Scanning Auger electron spectroscopy (AES) was performed in the friction test stylus tracks and in adjacent regions. X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and extended X-ray absorption fine structure (EXAFS) were selectively used.

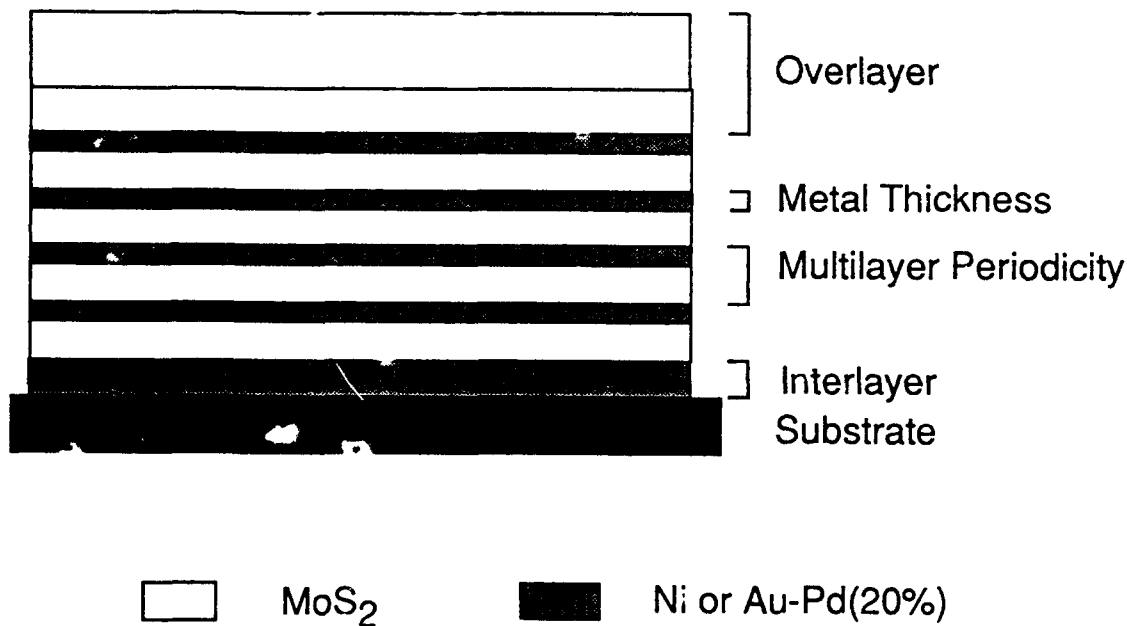


Figure 1. Schematic of multilayer lubricant film architecture.

III. RESULTS

A. PREFERRED ORIENTATION AND MORPHOLOGY OF FILMS

XRD confirmed that the presence of metal multilayers affects preferred orientation. The reference RF-AT film has a (100) [and (110); not shown here] preferred orientation without (002) basal orientation (Figure 2a). In contrast, the pure RFM film has mixed crystalline orientations, including basal (002) and edge (100) [and (110); not shown here] as shown in Figure 2b. Broad peaks were evident. The presence of 0.7 nm of Ni per 10 nm [effectively 3 monolayers (MLs) of Ni per 15 layers of MoS₂] resulted in a strong (002) preferred orientation as shown in Figure 2c. The 0.9nm-Au/10nm film was similar, as shown in Figure 2d. In both multilayer films third- and fourth-order multilayer peaks resulting from diffraction by the multilayers were detected. The diffraction peak locations confirmed the multilayer spacing. Decreasing the multilayer spacing from 10 to 5 nm also resulted in a strong (002) preferred orientation, as shown in Figure 2e for the 0.3nm-Ni/5.0nm film. However, thicker Ni multilayers at 5 nm spacing (2 to 4 MLs) resulted in suppression of the (002) peak, although higher order multilayer reflections become visible, as shown in Figures 2f through 2h. Although not shown here, the 0.2nm-Ni/3.3nm had basal plane preferred orientation while the 0.8nm-Ni/3.9nm and IBAD-prepared 1.5nm-Au/3.6nm films did not exhibit (002), (100) or (110) peaks.

The SEM revealed that the presence of multilayers densified and refined the film morphology. However, relative to the RF-AT film (Figure 3a) with the columnar plate structure, even the pure MoS₂ RFM films had a dense morphology with equiaxed features (Figure 3b). The dense morphology of the pure film is similar to that observed for other MoS₂ films grown at this pressure.^{4,12,13} The presence of 0.7nm-Ni/10nm yields a smoother morphology with discernable features on the fracture cross-sectional surface that are smaller in size relative to the pure RFM film (Figure 3c). A 0.9nm-Au/10nm film has a similar morphology to the 0.7nm-Ni/10nm film (Figure 3d). Note that the size of the equiaxed features in the multilayer films range from 20 to 60 nm, which exceeds the multilayer spacing of 10 nm. Reducing the spacing from 10 to 5 nm results in very smooth, dense morphologies (Figure 3e for 0.3nm-Ni or 1 ML Ni per 5 nm); higher metal thicknesses at 5 nm spacing yielded morphologies that were progressively smoother (Figures 3f through 3h). The 0.2nm-Ni/3.3nm, 0.8nm-Ni/3.9nm, and 1.5nm-Au/3.6nm films (not shown) also had smooth, dense morphologies.

B. WEAR AND INDENTATION TESTS

Thrust washer sliding tests indicate that pure RFM films had endurance equivalent to or better than the averaged AT film reference value (Figure 4). The presence of increasing thicknesses of Ni at 5 nm spacing reduced average endurance. However, the endurance values of the Ni multilayer films were quite scattered; many thicker Ni films failed instantly. Experience with this wear test suggested that the bare steel counterface was not receiving an adequate lubricant transfer film during the early stages of contact. Based on this hypothesis, the Group II films were made with a 50-nm overlayer of pure MoS₂ to facilitate transfer film formation. The 0.7nm-Ni/10nm films had endurance comparable to the RF-AT film average. Also shown are the 0.9nm-Au/10nm films, which had a somewhat lower endurance than the 0.7nm-Ni/10nm films. In contrast, the 1.6nm-Au/3.6nm films (prepared by IBAD) that did not possess pure MoS₂ overlayers had lifetimes that exceeded those of comparable high-Ni counterparts such as the 0.8nm-Ni or 1.0nm-Ni/5.0nm films. The films containing Au will be discussed further in Section IV.

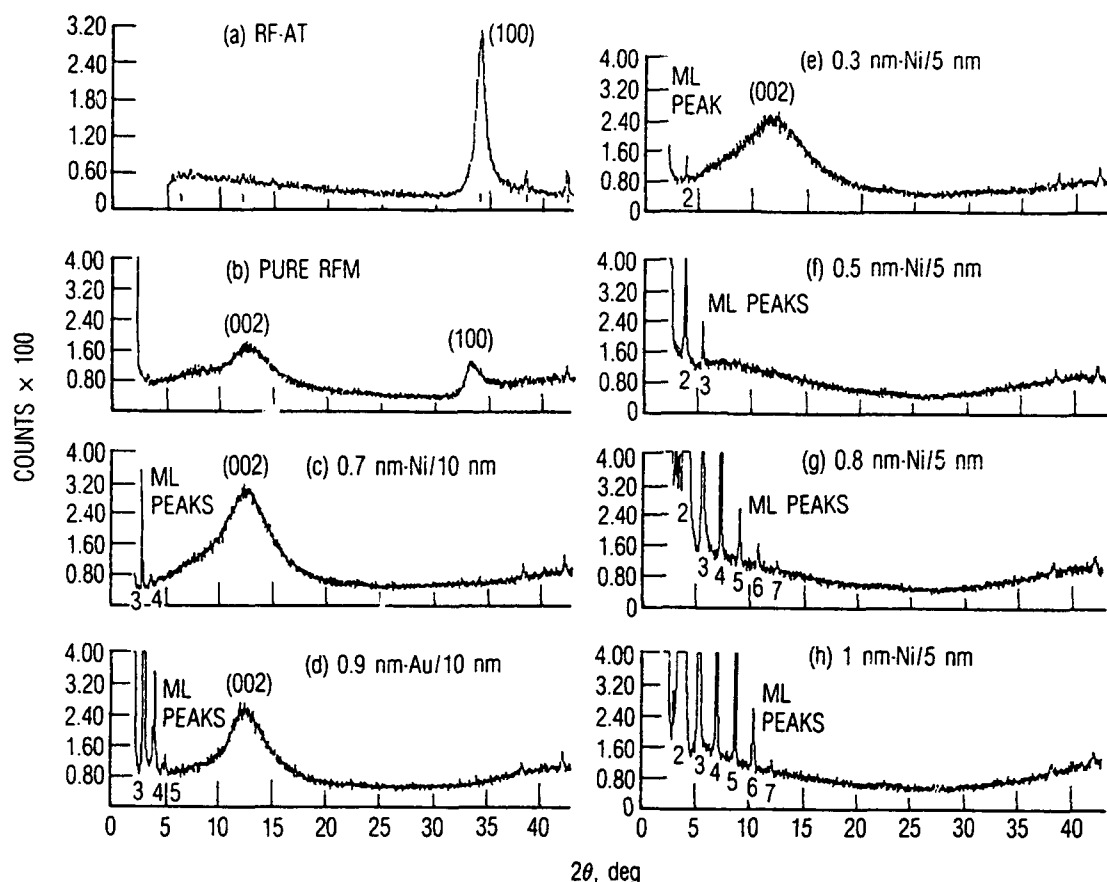
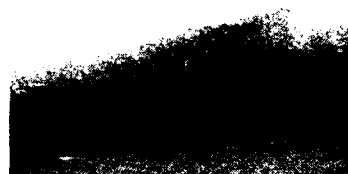


Figure 2. XRD scans of the MoS_2 films showing the preferred orientation and crystallinity of the films as a function of multilayer composition. (a) The pure MoS_2 RF-AT reference film has strong (100), and (110) [the latter is not shown] preferred orientation. (b) The pure MoS_2 RF magnetron film has poorly crystalline but mixed (002), (100), and (110) [the latter is not shown] preferred orientation. (c,d) The presence of metal multilayers, such as 0.7nm-Ni/10nm or 0.9nm-Au/10nm, suppresses edge orientations in favor of (002) basal plane orientation. (e) Thin 0.3-nm multilayers of Ni with 5 nm spacing also result in (002) basal plane preferred orientation. (f-h) At 5-nm spacing, increasing the thickness of the Ni multilayers suppresses the detected basal plane preferred orientation of the MoS_2 layers, while enhancing the detection of higher-order multilayer (ML) reflections. Unlabeled peaks represent the 440C steel substrate.



(a) RF-AT



(b) Pure RFM



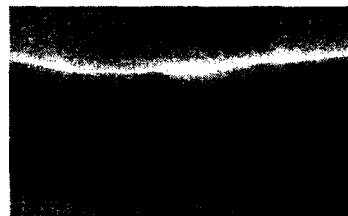
(c) 0.7 nm-Ni/10 nm



(d) 0.9 nm-Au/10 nm



(e) 0.3 nm-Ni/5 nm



(f) 0.5 nm-Ni/5 nm



(g) 0.8 nm-Ni/5 nm



(h) 1 nm-Ni/5 nm

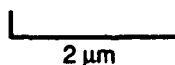


Figure 3. Cross-sectional SEM micrographs of the films shown in Figure 2. (a) The RF-AT reference film deposited at 2.66 Pa has the columnar-plate morphology. (b) The pure MoS_2 RF magnetron film, deposited at 0.266 Pa, has a dense, equiaxed morphology that is consistent with literature reports of films deposited at this pressure (see text). The increasing presence of metal multilayers refines the equiaxed features and yields progressively smoother films: (c) 0.7nm-Ni/10nm, (d) 0.9nm-Au/10nm, (e) 0.3nm-Ni/5nm, (f) 0.5nm-Ni/5nm, (g) 0.8nm-Ni/5nm, and (h) 1nm-Ni/5nm. (Sample tilt: (a) 30° , (b-h) 45° .)

The 52100 steel thrust bearing rolling data show scattered endurance values for the 5-nm spacing Ni films (Figure 5a), though the 0.3nm-Ni/5nm films had better endurance than the thicker Ni films. Some films failed extremely early. Ball/cage wear effects were found to have limited endurance in tests of the 0.2nm-Ni/3.3nm, 0.8nm-Ni/3.9nm, and 0.8nm-Ni/5nm films. Excessive ball/cage wear caused the cage to contact the raceway land (which was confirmed by visual examination following the test) causing increased torque. SEM examination revealed limited (i.e., patchy) removal of MoS₂ from the ball raceway contact tracks for these films. In contrast, all other films showed uniform film removal in the wear track region at end of life.

A different problem was observed for the pure RFM films deposited without a nickel interlayer on 52100 steel thrust bearings. The pure RFM films had poor adhesion to 52100 steel, i.e., partial delamination of MoS₂ was observed prior to testing. These films had endurance values less than 10% of those of the RF-AT films deposited onto 440C bearings. In contrast, pure MoS₂ films deposited on 440C steel with a Ni interlayer did not have adhesion problems and had greater endurance values than the RF-AT films (Figure 5b). (One of the pure RFM MoS₂ films on 440C had visible damage in the form of a piece of remnant adhesive tape in the raceway. The tape was removed prior to testing, exposing a small, < 1 mm², area of uncoated steel. This damaged film had a lower endurance than the other two bearings.) All of the sliding tests were run on 440C, and such adhesion problems were not observed for any films. The multilayer films on both steels had initial metal layers (either Ni or Au) between the first layer of MoS₂ and the substrate, which probably improved adhesion to the steel substrates, particularly for the 52100.

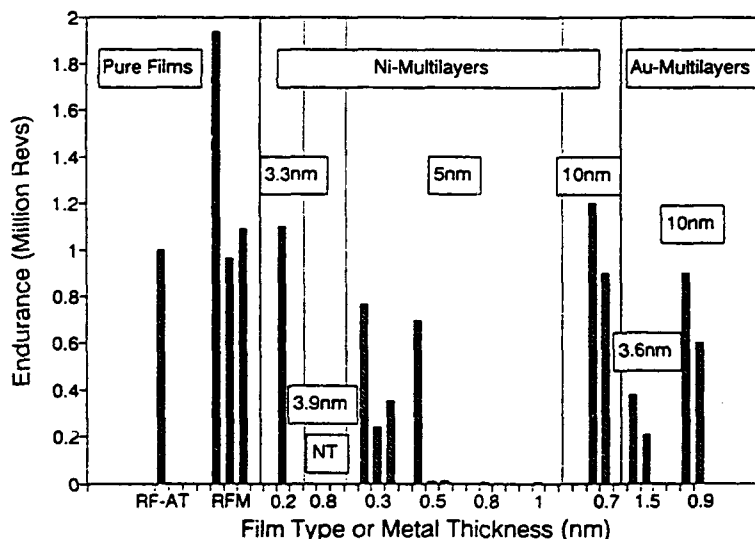


Figure 4. Endurance data for the various films in thrust washer sliding tests from Groups I and II. All substrates are 440C steel. The inset values represent the multilayer periodic spacing in nm. Increasing metal thickness at 5-nm spacing generally decreased average endurance. The pure RFM films (from Deposition Group I, without nickel interlayers [see text]) and 10-nm spacing multilayer films (from Group II) performed comparably to the RF-AT reference in sliding. (NT = not tested.)

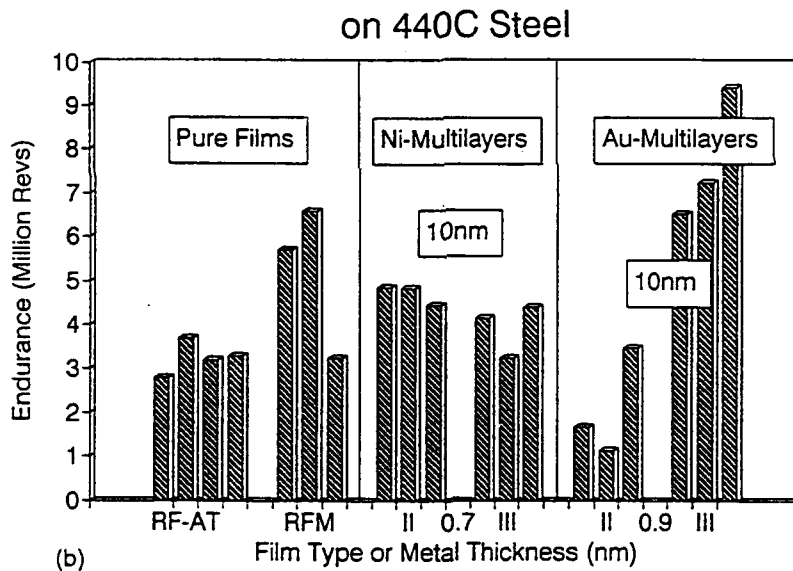
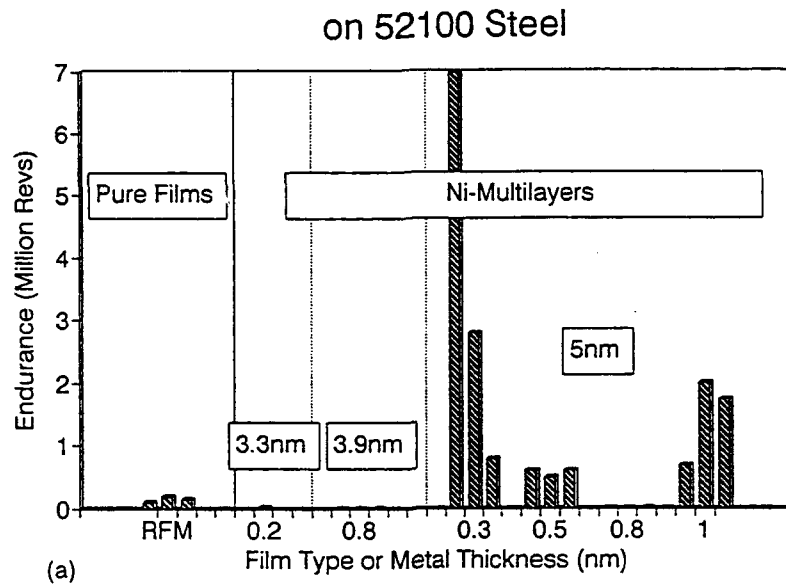


Figure 5. Endurance data from thrust bearing rolling tests of MoS_2 on: (a) 52100 steel (Deposition Group I, see text), and (b) 440C steel (Groups II and III). The inset values represent the multilayer periodic spacing in nm. Increasing metal thickness at 5-nm spacing generally decreased average endurance. The pure RFM films on 52100 steel had adhesion problems that limited endurance. The pure RFM films on 440C had excellent endurance, although one bearing had lower endurance. The film on this bearing had visible damage in the form of a remnant piece of adhesive tape at one small area in the raceway. The 0.7nm-Ni/10nm films consistently exceeded the performance of the RF-AT films. The 0.9nm-Au/10nm films from Group II had deposition irregularities (see text) that limited endurance. The deposition problems were rectified to prepare the Group III Au-containing films, which have the highest endurance of the films tested.

The 0.7nm-Ni/10nm films (with 50 nm pure MoS₂ overlayers) had endurance lives consistently exceeding those of the RF-AT films (Figure 5b). (Actual rolling endurance values are shown for the RF-AT films; the values are not averages). The 0.9nm-Au/10nm films had lower endurance than their Ni counterparts from Group II, following the trend in the sliding data. However, Group III 0.9nm-Au/10nm films showed improved performance over Group II Au-containing films. This improvement apparently resulted from better deposition plasma stability in Group III (see Section IV, DISCUSSION). Indeed, the 0.9nm-Au/10nm films have the best endurance values of all films tested. The Ni-containing multilayer films had equivalent endurance values between the Group II and III films.

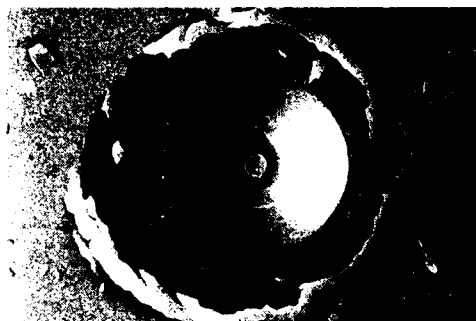
Brale indentation (150 kg load) resulted in film delamination at the coating-substrate interface for the pure films in a manner consistent with dense morphologies,¹¹ i.e., extensive delamination on the order of 200 to 300 μ m was observed. Increasing metal content in the multilayer films inhibited delamination (Figure 6), suggesting that the fracture toughness of the films had increased. Films with 10 nm spacing had more delamination than 5 nm films.

C. FRICTIONAL BEHAVIOR OF FILMS

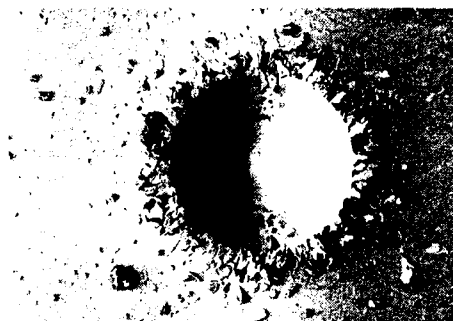
The friction coefficients determined in pin-on-disk tests as a function of number of cycles are shown in Figure 7 for five film preparations. The pure RFM film and the 0.9nm-Au/10nm film exhibited similar friction behavior in air and ultrahigh vacuum, with friction coefficients at 1000 cycles of 0.05 to 0.08. In air, the 0.7nm-Ni/10nm film had a higher friction coefficient than its gold-containing counterpart, despite the presence of a 50 nm MoS₂ surface layer in both cases. The 0.3nm-Ni/5nm and 0.8nm-Ni/3.9nm films showed increasing friction coefficient with increasing nickel layer thickness. In UHV, all structures except the 0.8nm-Ni/3.9 nm film had friction coefficients below 0.08. The 0.7nm-Ni/10nm and 0.3nm-Ni/5nm films had notably low coefficients in UHV, relative to air. The 0.3nm-Ni/5nm film performed well despite the lack of the extra 50 nm MoS₂ surface layer present in the larger spacing films. AES analysis of the wear tracks detected metal from the metal multilayers only in the 0.8nm-Ni/3.9nm film. In fact, iron was observed in addition to nickel in all wear track locations for this specimen in both test environments, suggesting that severe film spallation exposed the substrate. Oxygen content on the wear track surface decreased after 1000 cycles in air for this film while oxygen content on the surface increased after 1000 cycles for all other films. In UHV, surface oxygen content on the wear track decreased for all films.

D. FILM COMPOSITION

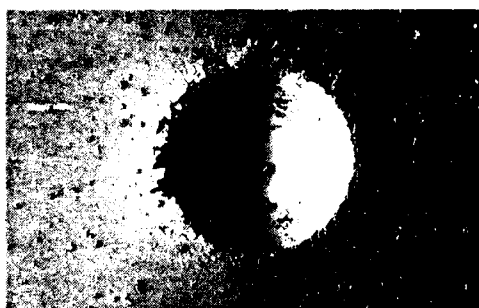
Preliminary characterization results of the multilayer films using AES, XPS, Raman spectroscopy, and EXAFS have revealed the following: (1) AES and XPS of as-prepared films indicate that the films are sulfur deficient relative to MoS₂ single crystals; the near-surface regions of the films are best described as MoS_x with x generally being in the range of 1.5 to 1.7. (2) XPS of the as-prepared films has determined that the ratio of Mo⁴⁺/(Mo⁴⁺+Mo⁶⁺) ranges from 0.70 to 0.76; no consistent trend was found as a function of multilayer composition. (3) Oxygen and carbon were detected on the surface of the as-prepared films, with the amount generally increasing with nickel content. (4) No Mo-O stretching vibrations (800-1000cm⁻¹ frequency range) were detected by Raman, as shown in Figure 8. (5) EXAFS studies on films using Kapton substrates revealed the presence of considerable Ni-O bonding and virtually no Ni-Mo or Ni-S bonding in the nickel multilayer films.²¹



(a) Pure RFM



(b) 0.8 nm-Ni/5 nm



(c) 1 nm-Ni/5 nm



(d) 0.7 nm-Ni/10 nm



0.5 mm

Figure 6. SEM micrographs of delamination at the film-substrate interface following Brale indentation of selected films. (a) The pure RFM film delaminates extensively. (b-c) The presence of increasing metal content, 0.8nm-Ni/5nm and 1nm-Ni/5nm, inhibits delamination. (d) Increasing the multilayer spacing (0.7nm-Ni/10nm) increases delamination relative to the 5-nm spacing films.

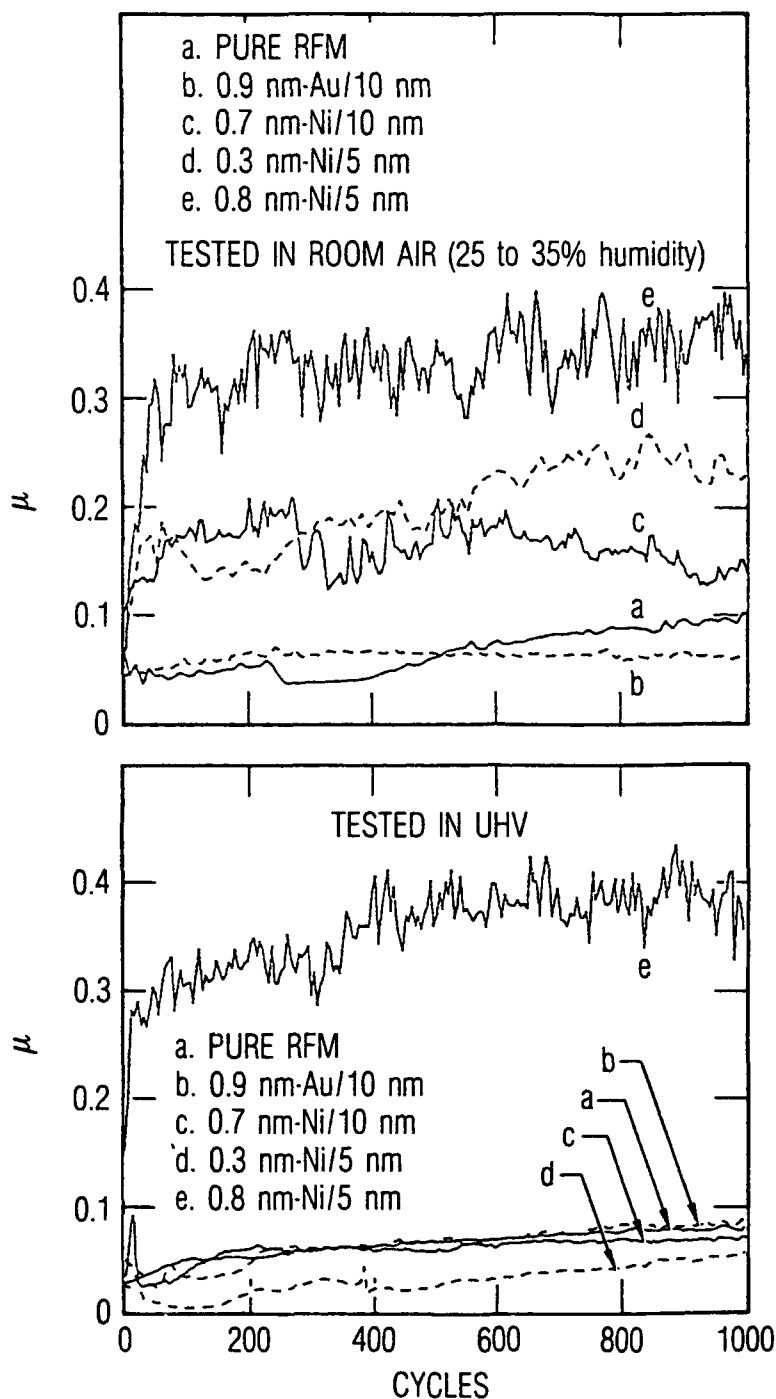


Figure 7. Pin-on-disk friction coefficients versus number of disk revolutions for sputtered MoS_2 films in air with 25 to 35% relative humidity (top graph), and in ultrahigh vacuum (bottom graph). All tests were run at 1 GPa maximum contact stress and 3.5 to 5.5 cm/s sliding velocity. (a) RFM pure MoS_2 , (b) 0.9nm-Au/10nm (c) 0.7nm-Ni/10nm, (d) 0.3nm-Ni/5nm, and (e) 0.8nm-Ni/3.9nm. Films (b) and (c) had a 50-nm overlayer of pure MoS_2 .

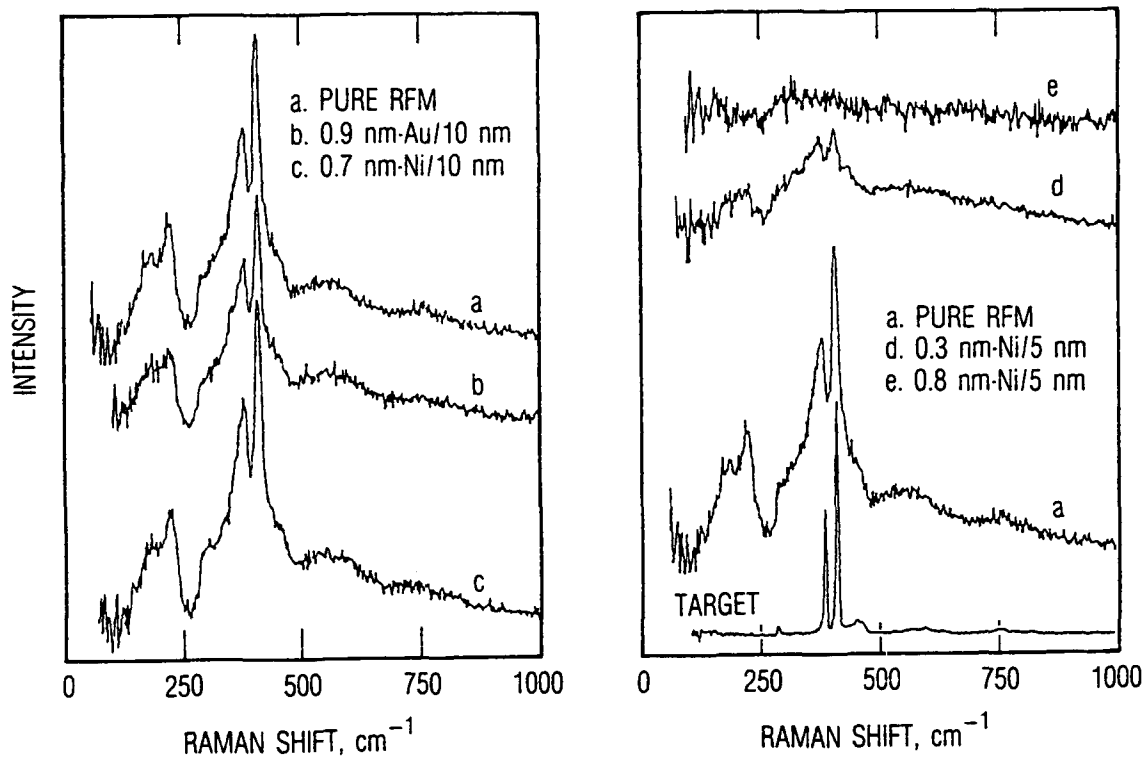


Figure 8. Raman spectra for (a) pure RFM film, (b) 0.9nm-Au/10nm film, (c) 0.7nm-Ni/10nm, (d) 0.3nm-Ni/5nm, and (e) 0.8nm-Ni/3.9nm. A Raman spectrum in the same frequency range for a MoS₂ sputtering target is shown for comparison. The first order bands for MoS₂ shown are at 286 cm⁻¹ (E_{1g}, S-S basal plane), 383 cm⁻¹ (E_{2g}, Mo-S basal plane), and 408 cm⁻¹ (A_{1g}, S-S c-axis). Of particular note is the absence of bands from Mo-O stretching vibrations, which would be expected in the 800 to 1000 cm⁻¹ frequency range. The depth sensitivity of Raman is approximately 200 nm for the pure RFM film. The presence of metal in the multilayer films makes them more insensitive to Raman by increasing reflectivity.

IV. DISCUSSION

The experimental data verify the hypothesis that metal multilayers, of appropriate spacing and thickness, can promote the formation of sputter-deposited MoS₂ with essentially exclusive basal plane orientation and low porosity. We propose that the ability of multilayers to cause preferred orientation exploits the early-growth behavior of sputter-deposited MoS₂ films. Near the interface, i.e., < 20 nm thick, sputter-deposited MoS₂ films often have a very low (in some cases zero) areal ratio of edge islands to basal islands present. For example, we previously found that on carbon substrates, edge island presence was not significant until a thickness of 25 to 40 nm had been deposited for films deposited at 70 °C (AT) and at 220°C (HT).⁸ Careful XRD studies of MoS₂ grown on 440C steel suggest a similar behavior. Bertrand has found by XRD that edge orientation can be removed in thin films grown on Si or Au by heating the substrate (>100°C) prior to or during deposition.⁷ In a cross-sectional TEM study of rf-magnetron sputtered MoS₂ grown on Si at 300°C (effectively duplicating the deposition conditions in the work by Bertrand), Moser et al. have found that highly crystalline basal orientation is confined to the first 10 to 20 nm near the interface. Poorly crystalline edge orientation develops above the basal layer, apparently from growth defects and basal plane crystal curvature in the MoS₂.¹⁸ We believe that deposition of metal multilayers continually reimposes new surfaces upon which sputter-deposited MoS₂ must nucleate. This newly nucleated MoS₂ on the film surface has a high (or exclusive) degree of basal orientation. A new metal layer is then deposited before any significant edge orientation can develop that might lead to competitive growth, columnar morphology, and porosity.

The structures of the metal-MoS₂ interfaces of the multilayer films require further study. Close examination of the XRD patterns in Figures 2c and 2g, representing the 0.7nm-Ni/10nm and 0.8nm-Ni/5nm films, respectively, suggests that poorly crystalline regions exist near the metal layers. These poorly crystalline regions or layers may contain misaligned, curved, or buckled MoS₂ basal planes. Roughness or waviness of the metal layers may cause these crystal distortions. The continuity of the metal layers also needs to be determined.

The above interpretation of growth suggests some limits, in terms of interlayer thickness and spacing, to obtaining basal orientation. The minimum metal thickness tested, which was 0.2 nm or ≈1 ML of Ni, caused basal orientation to develop. TEM studies of co-sputtered films suggest (if one assumes that the percentage of dopant found in the film after deposition equals the fractional flux of dopant during deposition) that the Ni layer could be thinner, perhaps 0.02-0.06 nm, and still suppress edge growth.¹⁷ However, from a multilayer production and quality control viewpoint 0.5 to 1.0 nm (two to four monolayers) layers are more optimum metal thicknesses. Metal multilayers of these thicknesses are easier to reproduce and readily allow XRD to be performed to measure, via the multilayer peaks, actual multilayer spacing (and thus total film thickness because the number of multilayers is known). Multilayer quality can also be further assessed via peak shape analysis.¹⁹

The wear data show that there are some optimum combinations of multilayer spacing and composition to obtain favorable tribological properties. For the Ni multilayer films, better endurance was achieved with 10 nm spacing than with 5 nm. We interpret the data to mean that once basal orientation is obtained by imposition of a metal multilayer, the best tribological performance is achieved by depositing the maximum amount possible of MoS₂ with basal orientation between metal layers. (In principle, the best storage oxidation resistance would be obtained by films having this preferred orientation because oxidation is slow through basal- oriented films.²²) Thus, the upper limit on spacing might be the criti-

cal thickness at which growth defects, such as basal plane curvature, induce or allow edge orientation to develop. Cross-sectional¹⁸ and plan-view⁸ TEM studies of pure MoS₂ films suggest that this upper limit is in the range between 10 to 25 nm. A top layer of pure MoS₂ over the multilayer film appears to help form lubricant transfer films on bare counterfaces. Further work is needed to determine optimum top layer thicknesses. A pure MoS₂ overlayer may significantly improve the performance of the 5 nm spacing Ni films; the possibility merits further study.

The friction coefficients measured for most of the films in UHV, ranging between 0.04 and 0.08, are consistent with films having this dense morphology and sulfur deficiency reported by Nabot et al., who measured coefficients in the range of 0.04 to 0.06.¹³ The high friction coefficient of the 0.8nm-Ni/3.9nm film and the presence of iron in the wear track suggests that catastrophic failure had occurred. The high concentration of Ni promotes seizure instead of lubrication. The poor endurance of the 0.5-1.0nm-Ni/5nm films in the wear tests suggests that seizure also occurred in those tests.

Further composition investigations of the multilayer films are needed to determine the distribution of elements and their bonding in these obviously heterogeneous structures. In particular, detailed depth profile studies and also EXAFS of the films could elucidate the bonding of oxygen in these films. Oxygen apparently bonds to nickel in the films. The Mo⁶⁺ detected by XPS is probably on the film surface since Raman data did not reveal vibrations representing molybdenum oxides deeper in the film bulk. Oxygen may also be substitutionally present as MoS_{2-x}O_x and would be difficult to identify by Raman. In this phase, oxygen could contribute to good film lubricity by expanding the basal plane separation as discussed elsewhere.^{23,24}

We had hypothesized that Au, being softer and more inert than Ni (particularly to oxidation), might be a better constituent in a multilayer lubricant film, but the initial wear data (from Group II) showed that the 0.9nm-Au/10nm films had lower endurance with higher scatter than the corresponding Ni (0.7nm-Ni/10nm) films, while later tests (from Group III) showed improved, superior performance for the Au-containing films. These differences are explained by the higher sputtering rate of Au versus Ni, which necessitated a lower power setting for the Au films in Group II that was unstable--visible plasma pulsations were observed. Higher MoS₂ growth rates were obtained in Group III by using two MoS₂ targets with one Au target to avoid this problem of irregular deposition by allowing the Au target to be operated in a higher, stable power range. The IBAD 1.5nm-Au/3.6nm films also had far higher endurance than their sputter-deposited Ni counterparts (0.5-1.0nm-Ni/5 nm). Thus, it appears that the presence of Au in the multilayer films improves endurance relative to either Ni-MoS₂ multilayer or pure MoS₂ films.

As mentioned earlier, dense morphologies are desirable to prevent conversion of a large fraction of the total film thickness into film debris early in wear, and would be suitable, for example, in precision bearings. Both the multilayer and pure RFM films prepared in this study had dense morphologies. The dense morphology of the pure films agrees with literature reports of films deposited at 0.266 Pa. However, the multilayer films of appropriate metal thickness and spacing offer several advantages: (1) the basal plane preferred orientation is in the optimum orientation for lubrication; (2) the multilayer structure allows XRD to provide a non-destructive quality control determination of film thickness that is critical for precision bearing applications; (3) the basal plane preferred orientation should offer maximum oxidation resistance (a benefit because spacecraft are often stored before flight)²²; (4) the metal layers, if proper elements are selected, may also contribute to oxidation resistance; and (5) multilayer composition (especially Au layers) could affect electrical conductivity in sliding electrical contacts.

V. CONCLUSIONS

The deposition of metal multilayers of appropriate layer thickness and periodic spacing in sputter-deposited MoS₂ solid lubricant films can induce exclusive (002) basal plane preferred orientation. The orientation results can be explained in terms of a model based on active sites and competitive growth. The films possessed dense, equiaxed or smooth morphologies. Columnar growth was inhibited in part by the deposition pressure used, although the presence of multilayers resulted in smoother morphologies. Some multilayer films exhibited excellent endurance in sliding wear and thrust bearing tests.

In this study, the optimum structures appear to have the following characteristics: (1) large multilayer spacings, on the order of 10 nm, that contain MoS₂ with exclusive basal plane orientation between the metal layers, and (2) a minimum metal layer thickness that generates strong multilayer XRD diffraction peaks for quality control assessment (i.e., 0.5 to 1.0 nm) and induces basal orientation. A thin surface overlayer of pure MoS₂ appeared to facilitate transfer film formation on uncoated counterfaces. Friction coefficients in UHV ranged between 0.05 and 0.08. These values seem to stem in part from the film morphology and also from apparent surface sulfur sub-stoichiometry, i.e., in the range of $x = 1.5-1.7$ for MoS_x. Raman spectroscopy did not detect molybdenum oxides in the film bulk. The nature of oxygen bonding in the multilayer films and their storage resistance to oxidation require further study.

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